≋USGS

science for a changing world

Techniques of Water-Resources Investigations
of the United States Geological Survey

Chapter A1

METHODS FOR DETERMINATION
OF INORGANIC SUBSTANCES
IN WATER
AND FLUVIAL SEDIMENTS

By Marvin J. Fishman and Linda C. Friedman, Editors

First Edition 1970
Second Edition 1979
Third Edition 1989

Book 5
LABORATORY ANALYSIS

Mercury, atomic absorption spectrometric, flameless

Parameters and Codes:
Mercury, dissolved, I-1462-85 (μg/L as Hg): 71890
Mercury, total recoverable, I-3462-85 (μg/L as Hg): 71900
Mercury, suspended recoverable, I-7462-85 (μg/L as Hg): 71895
Mercury, recoverable-from-bottom-material, dry wt, I-5462-85 (μg/g as Hg): 71921

1. Application

1.1 This method may be used to analyze water and water-suspended sediment containing at least 0.5 μg/L of mercury. Samples containing mercury concentrations greater than 10 μg/L need to be diluted. Industrial and sewage effluent may be analyzed, as well as samples of fresh and saline water.

1.2 Suspended recoverable mercury is calculated by subtracting dissolved mercury from total recoverable mercury.

1.3 This method may be used to analyze bottom material containing at least 0.01 μg/g of mercury. Usually, a 5-g sample of prepared material (method P-0520) is taken for analysis. For samples containing more than 1.0 μg/g, use less sediment.

1.4 Total recoverable mercury in water-suspended sediment may be determined after each sample has been thoroughly mixed by vigorous shaking and a suitable sample portion has been rapidly withdrawn from the mixture.

2. Summary of method

2.1 The cold-vapor, flameless, atomic absorption procedure is based on the absorption of radiation at 253.7 nm by mercury vapor. Organic mercury compounds, if present, are decomposed by hot (95 °C) digestion with potassium permanganate and potassium persulfate in acid solution. The mercuric ions are then reduced to the elemental state with stannous chloride, and the mercury vapor is subsequently removed from solution by aeration and passed through a cell positioned in the light path of an atomic absorption spectrometer.

2.2 This method is based on a procedure described by Hatch and Ott (1968) and is similar in substance to the flameless atomic absorption method in "Methods for Chemical Analysis of Water and Wastes," published by the Water Quality Office of the Environmental Protection Agency (1979).

3. Interferences

3.1 Some samples may contain volatile organic compounds that absorb radiation at 253.7 nm and that may be swept from the solution along with the mercury vapor. These constitute a positive interference, and the possibility of their presence must not be overlooked.

3.2 Selenium concentrations, either as selenate or selenite, up to 10,000 μg/L do not interfere; higher concentrations were not tested.

4. Apparatus

4.1 *Absorption cell* (fig. 29). Mount and align an absorption cell (10- to 20-cm path length) in the light path of the spectrometer. Position a 60-watt lamp over the cell (10 to 15 cm) to prevent condensation of water vapor. Attach a sufficient length of tubing to the outlet of the cell and vent to a hood. Connect the inlet of the cell to the aerator with a minimum length of plastic tubing. Attach a water aspirator to the outlet of the stopcock (NOTE 1). Alternately, a forced-air pump may be used.

NOTE 1. The stopcock must remain closed during analysis and be opened only briefly between samples to remove residual mercury vapor from the absorption cell.
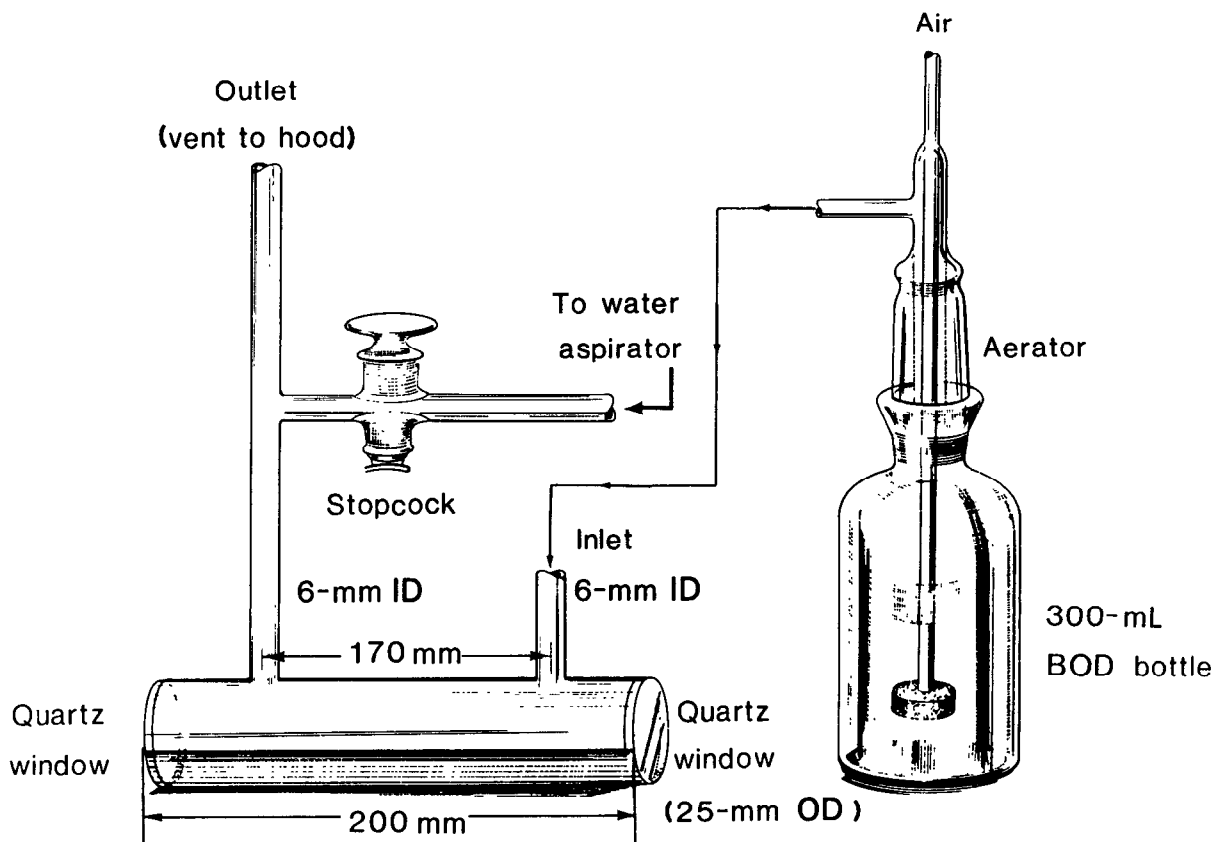
4.2 *Aerator* (fig. 29).

Figure 29.—Absorption cell and aerator

4.3 *Atomic absorption spectrometer and recorder* or a commerical mercury analyzer.

4.4 Refer to the manufacturer's manual to optimize instrument for the following:

Grating . . . . . . Ultraviolet
Wavelength . . 253.7 nm
Source . . . . Mercury-vapor dis-
charge, hollow-
cathode, or electrode-
less-discharge lamp

4.5 *BOD bottle*, 300-mL capacity.

4.6 *Water bath* or controlled-temperature oven, 95 °C.

5. Reagents

5.1 *Hydroxylamine hydrochloride-sodium chloride solution*: Dissolve 10 g NH₂OH·HCl and 12 g NaCl in demineralized water and dilute to 100 mL (NOTE 2). Prepare fresh daily. Alternatively, 12 g hydroxylamine sulfate may be used instead of the hydroxylamine hydrochloride.

NOTE 2. A larger volume of this reagent can be prepared if it is kept refrigerated.

5.2 *Mercury standard solution I*, 1.00 mL = 100 μg Hg: Dissolve 0.1712 g $Hg(NO_3)_2 \cdot H_2O$ in demineralized water. Add 1.5 mL concentrated HNO_3 and dilute to 1,000 mL with demineralized water.

5.3 *Mercury standard solution II*, 1.00 mL = 1.00 μg Hg: Dilute 5.00 mL mercury standard solution I and 1.5 mL concentrated HNO_3 to 500 mL with demineralized water. This and the following mercury standard solutions must be prepared fresh daily.

5.4 *Mercury standard solution III*, 1.00 mL = 0.050 μg Hg: Dilute 10.0 mL mercury standard solution II and 1.5 mL concentrated HNO_3 to 200 mL with demineralized water. Use this solution to prepare working standards at the time of analysis.

5.5 *Nitric acid*, concentrated (sp gr 1.41), with low mercury content: duPont, reagent-grade acid has been found satisfactory.

5.6 *Potassium permanganate solution*, 50 g/L: Dissolve 5 g $KMnO_4$ in demineralized water and dilute to 100 mL. Prepare fresh weekly. Store in brown glass bottle.

5.7 *Potassium persulfate solution*, 50 g/L: Dissolve 5 g $K_2S_2O_8$ in demineralized water and dilute to 100 mL.

5.8 *Stannous chloride solution*, 74 g/L: Add 22 g $SnCl_2 \cdot 2H_2O$ to 250 mL 0.25M H_2SO_4. This solution is unstable. Prepare fresh daily.

5.9 *Sulfuric acid*, concentrated (sp gr 1.84).

5.10 *Sulfuric acid*, 0.25M: *Cautiously* add 14 mL concentrated H_2SO_4 (sp gr 1.84) to demineralized water and dilute to 1 L.

6. Procedure

6.1 Immediately before each use, clean all glassware used in this determination by rinsing, first with warm, dilute HNO_3 (1+4), and then with demineralized water.

6.2 Follow instructions in paragraphs 6.2.1 through 6.2.5 for dissolved mercury.

6.2.1 Pipet a volume of sample containing less than 1.0 μg Hg (100 mL max) into a 300-mL capacity BOD bottle and adjust the volume to approx 100 mL.

6.2.2 Prepare a blank of demineralized water and sufficient standards, and adjust the volume of each to approx 100 mL with demineralized water.

6.2.3 Add 5 mL concentrated H_2SO_4 and 2.5 mL concentrated HNO_3, mixing after each addition.

6.2.4 Add 5 mL $KMnO_4$ solution and shake. Add additional small amounts of $KMnO_4$ solution, if necessary, until the purple color persists for at least 15 min.

6.2.5 Add 2 mL $K_2S_2O_8$ solution, mix, and heat for 2 h in a boiling-water bath or for 3 h in an oven at 95 °C. Proceed to paragraph 6.5.

6.3 Follow instructions in paragraph 6.3.1 through 6.3.5 for total recoverable mercury.

6.3.1 Pipet a volume of well-mixed sample containing less than 1.0 μg Hg (100 mL max) into a 300-mL capacity BOD bottle and adjust the volume to approx 100 mL.

6.3.2 Prepare a blank of demineralized water and sufficient standards, and adjust the volume of each to approx 100 mL with demineralized water.

6.3.3 Add 5 mL concentrated H_2SO_4 and 2.5 mL concentrated HNO_3, mixing after each addition.

6.3.4 Add 15 mL $KMnO_4$ solution and shake. Add additional small amounts of $KMnO_4$ solution, if necessary, until the purple color persists for at least 15 min.

6.3.5 Add 8 mL $K_2S_2O_8$ solution, mix, and heat for 2 h in a boiling-water bath or for 3 h in an oven at 95 °C. Proceed to paragraph 6.5.

6.4 Follow instructions in paragraphs 6.4.1 through 6.4.5 for recoverable mercury from bottom material.

6.4.1 Place a weighed portion of sample containing less than 1.0 μg Hg (5 g max) into a 300-mL capacity BOD bottle and add approx 100 mL demineralized water.

6.4.2 Prepare a blank of demineralized water and sufficient standards, and adjust the volume of each to approx 100 mL with demineralized water.

6.4.3 Add 5 mL concentrated H_2SO_4 and 2.5 mL concentrated HNO_3, mixing after each addition.

6.4.4 Add 15 mL $KMnO_4$ solution and shake. Add additional small amounts of $KMnO_4$ solution, if necessary, until the purple color persists for at least 15 min.

6.4.5 Add 8 mL $K_2S_2O_8$ solution, mix, and heat for 2 h in a boiling-water bath or for 3 h in an oven at 95 °C. Proceed to paragraph 6.5.

6.5 Remove from water bath, cool, and add $NH_2OH \cdot HCl$-NaCl solution in 2-mL increments to reduce the excess permanganate, as evidenced by the disappearance of the permanganate color.

6.6 Add 5 mL $SnCl_2$ solution to one sample and immediately attach the bottle to the aerator (NOTE 3). Record the maximum absorbance. After maximum absorbance has been recorded, remove the BOD bottle and open the stopcock to the vacuum. Momentarily pinch off the vent tube in order to remove residual mercury vapor from the absorption cell. Treat each succeeding sample, blank, and standard in a like manner. NOTE 3. Use the atomic absorption compressed-air supply or a peristaltic pump to aerate the sample. Adjust the rate of air flow to approx 2 L/min.

7. Calculations

7.1 Determine the micrograms of mercury in the sample from a plot of absorbances of standards. Exact reproducibility is not obtained, and an analytical curve must be prepared with each set of samples.

7.2 Determine the concentration of dissolved or total recoverable mercury in each sample as follows:

$$\text{Hg } (\mu g/L) = \mu g \text{ Hg} \times \frac{1,000}{\text{mL sample aliquot}}$$

7.3 To determine the concentration of suspended recoverable mercury, subtract dissolved-mercury concentration from total-recoverable-mercury concentration.

7.4 Determine the concentration of mercury in air-dried bottom material as follows:

$$\text{Hg } (\mu g/g) = \frac{\mu g \text{ Hg in sample}}{\text{wt of sample (g)}}$$

8. Report

8.1 Report mercury, dissolved (71890), total-recoverable (71900), and suspended- recoverable (71895), concentrations as follows: less than 10 $\mu g/L$ and greater than or equal to 0.5 $\mu g/L$, nearest 0.1 $\mu g/L$; 10 $\mu g/L$ and above, two significant figures.

8.2 Report mercury, recoverable-from-bottom-material (71921), concentrations as follows: less than 1.00 $\mu g/g$, nearest 0.01 $\mu g/g$; 1.0 $\mu g/g$ and above, two significant figures.

9. Precision

9.1 Precision for dissolved mercury for 23 samples within the range of 0.3 to 15.5 $\mu g/L$ may be expressed as follows:

$$S_T = 0.152X + 0.164$$

where

S_T = overall precision, micrograms per liter, and

X = concentration of mercury, micrograms per liter.

The correlation coefficient is 0.9229.

9.2 Precision for dissolved mercury for six of the 23 samples expressed in terms of the percent relative standard deviation is as follows:

Number of laboratories	Mean ($\mu g/L$)	Relative standard deviation (percent)
22	0.33	50
15	.60	46
27	1.69	32
7	3.46	30
7	6.54	18
26	15.5	16

9.3 It is estimated that the percent relative standard deviation for total recoverable and suspended recoverable mercury and for recoverable mercury in bottom material will be greater than that reported for dissolved mercury.

9.4 Precision for total recoverable mercury expressed in terms of percent relative standard deviation for two water-suspended sediment samples is as follows:

Number of laboratories	Mean ($\mu g/L$)	Relative standard deviation (percent)
19	9.2	32
18	11.4	38

References

Fishman, M. J., and Brown, Eugene, 1976, Selected methods of the U.S. Geological Survey for the analysis of wastewaters: U.S. Geological Survey Open-File Report 76-177, p. 51-60.

Hatch, W. R., and Ott, W. L., 1968, Determination of submicrogram quantities of mercury by atomic absorption spectrophotometry: Analytical Chemistry, v. 40, p. 2085-90.

U.S. Environmental Protection Agency, 1979, Methods for chemical analysis of water and wastes: Cincinnati, p. 245.1-1.

Mercury, atomic absorption spectrometric, flameless automated-sequential

Parameter and Code:
Mercury, dissolved, I-2462-85 (μg/L as Hg): 71890

1. Application

This method may be used to analyze water and wastewater containing at least 0.1 μg/L mercury. Samples containing mercury concentrations greater than 8.0 μg/L need to be diluted.

2. Summary of method

2.1 The cold-vapor, flameless, atomic absorption procedure is based on the absorption of radiation at 253.7 nm by mercury vapor. Organic mercury compounds, if present, are decomposed by hot (95 °C) digestion with potassium dichromate and potassium persulfate in acid solution. Mercuric ions are then reduced to the elemental state with stannous chloride, and mercury vapor is subsequently removed from solution by aeration and passed through a cell positioned in the light path of an atomic absorption spectrometer.

2.2 The method is based on a procedure described by El-Awady and others (1976).

3. Interferences

3.1 Chloride concentrations up to 5,000 mg/L do not interfere; higher concentrations were not tested.

3.2 Hydroxylamine hydrochloride-sodium chloride solution is added to prevent interference from residual chlorine.

3.3 El-Awady and others (1976) reported that copper sulfate (1,000 mg/L) does not interfere and that chemical-oxygen-demand (COD) concentrations of less than 700 mg/L can be tolerated. Ethyl alcohol, methyl alcohol, glycerol, chloroform, and carbon tetrachloride did not interfere when added in concentrations as high as 0.5 percent. Major interferences were observed from benzene and toluene. A maximum tolerance of 500 μg/L was obtained for these compounds.

3.4 Selenate concentrations up to 10,000 μg/L do not interfere; higher concentrations were not tested. Concentrations of selenite greater than 100 μg/L interfere by suppressing the mercury absorption.

4. Apparatus

4.1 *Absorption cell*, 100-mm long, 10-mm diameter, with quartz windows.

4.2 *Atomic absorption spectrometer and recorder* or a commercial mercury analyzer.

4.3 Refer to manufacturer's manual to optimize instrument for the following:

Grating . . . . . . Ultraviolet
Wavelength
   counter . . . . 253.7 nm
Source . . . . Mercury-vapor, hollow-cathode, or electrodeless-discharge lamp
Carrier . . . . . . Nitrogen (flow approx 20 mL/min; adjust for maximum sensitivity with use of a standard)

4.4 *Technicon Autoanalyzer*, consisting of sampler, manifold (fig. 30), proportioning pump, and high-temperature heating bath (NOTE 1).

NOTE 1. Nitrogen gas may be used instead of air in manifold (fig. 30).

4.5 *Vapor-liquid separator* (fig. 31).

5. Reagents

5.1 *Hydroxylamine hydrochloride-sodium chloride solution*: Dissolve 30 g $NH_2OH \cdot HCl$ and 30 g NaCl in demineralized water and dilute to 1 L.
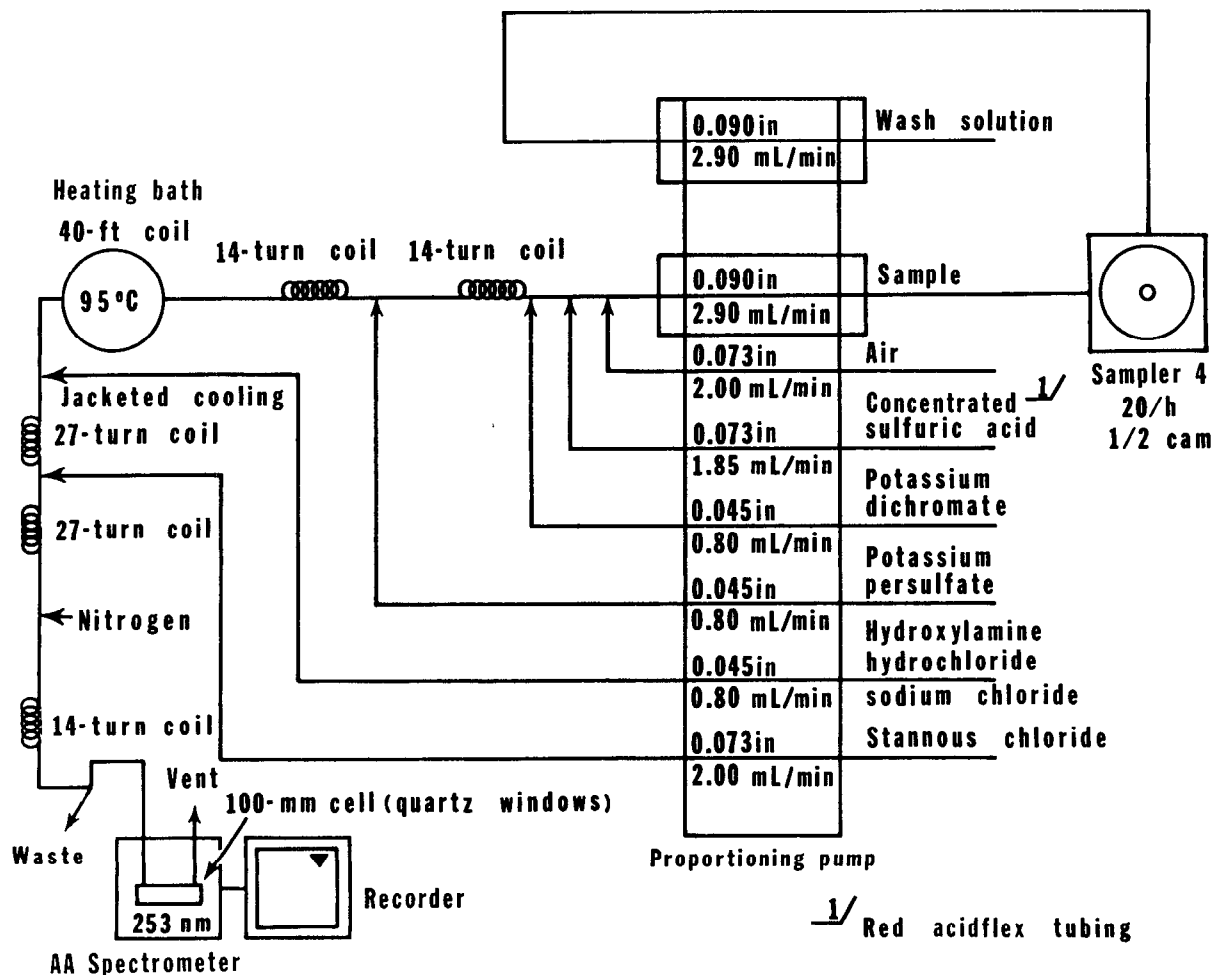
Figure 30.—Mercury manifold

Figure 31.—Vapor-liquid separator
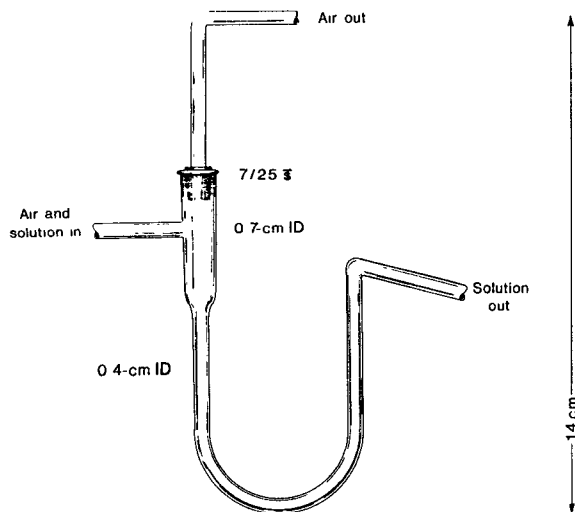
5.2 *Mercury standard solution I*, 1.00 mL = 100 μg Hg: Dissolve 0.1712 g Hg(NO$_3$)$_2$·H$_2$O in demineralized water. Add 1.5 mL concentrated HNO$_3$ (sp gr 1.41) and 25 mL K$_2$Cr$_2$O$_7$ solution, and dilute to 1,000 mL with demineralized water.

5.3 *Mercury standard solution II*, 1.00 mL = 1.00 μg Hg: Dilute 10.0 mL mercury standard solution I, 5 mL concentrated HNO$_3$, and 25 mL K$_2$Cr$_2$O$_7$ solution to 1,000 mL with demineralized water.

5.4 *Mercury standard solution III*, 1.00 mL = 0.100 μg Hg: Dilute 100.0 mL mercury standard solution II, 5 mL concentrated HNO$_3$, and 25 mL K$_2$Cr$_2$O$_7$ solution to 1,000 mL with demineralized water. Use this solution to prepare working standards. The working standards

should also contain 5 mL/L of concentrated HNO_3 and 25 mL/L of $K_2Cr_2O_7$ solution. The working standards are stable for at least 3 weeks.

5.5 *Nitric acid*, concentrated (sp gr 1.41), with low mercury content: Both duPont and Baker reagent-grade acids have been found satisfactory.

5.6 *Nitric acid (1+99)*, wash solution: Dilute 10 mL of concentrated HNO_3 (sp gr 1.41) to 1 L with demineralized water.

5.7 *Potassium dichromate solution*, 20 g/L: Dissolve 20 g $K_2Cr_2O_7$ in demineralized water and dilute to 1 L.

5.8 *Potassium persulfate solution*, 40 g/L: Dissolve 40 g $K_2S_2O_8$ in demineralized water and dilute to 1 L. Prepare fresh each week.

5.9 *Stannous chloride solution*, 84 g/L: Dissolve 100 g $SnCl \cdot 2H_2O$ in 100 mL concentrated hydrochloric acid (sp gr 1.19) and dilute to 1 L with demineralized water. This solution is unstable. Prepare fresh daily.

5.10 *Sulfuric acid*, concentrated (sp gr 1.84).

6. Procedure

6.1 Set up manifold (fig. 30).

6.2 Prepare a blank of demineralized water and sufficient standards to 8.0 μg/L by appropriate dilutions of mercury standard solution III.

6.3 Initially feed all reagents through the system using the nitric acid wash solution in the sample line. Allow the heating bath to warm to 95 °C.

6.4 Place a complete set of standards and a blank in the first positions of the first sample tray, beginning with the most concentrated standard. Place individual standards of differing concentrations in approximately every eighth position of the remainder of this and subsequent sample trays. Fill remainder of each tray with unknown samples.

6.5 Remove the sample line from the nitric acid wash solution when the baseline stabilizes and begin analysis.

7. Calculations

7.1 Prepare an analytical curve by plotting the absorbance of each standard versus its respective mercury concentration.

7.2 Compute the concentration of mercury in each sample by comparing its absorbance to the analytical curve. Any baseline drift that may occur must be taken into account when computing the absorbance of a sample or standard.

8. Report

Report mercury, dissolved (71890), concentrations as follows: less than 10 μg/L, nearest 0.1 μg/L; 10 μg/L and above, two significant figures.

9. Precision

9.1 Precision for dissolved mercury for 11 samples within the range of 0.3 to 16.4 μg/L may be expressed as follows:

$$S_T = 0.134X + 0.106$$

where

S_T = overall precision, micrograms per liter, and

X = concentration of mercury, micrograms per liter.

The correlation coefficient is 0.9722.

9.2 Precision for dissolved mercury for five of the 11 samples expressed in terms of percent relative standard deviation is as follows:

Number of laboratories	Mean (μg/L)	Relative standard deviation (percent)
3	0.33	17
4	.58	54
3	1.87	32
3	2.80	27
5	16.4	14

Reference

El-Awady, A. A., Miller, R. B., and Carter, M. J., 1976, Automated method for the determination of total and inorganic mercury in water and waste-water samples: Analytical Chemistry, v. 48, p. 110-16.

Molybdenum, atomic absorption spectrometric, chelation-extraction

1. Application

1.1 This method may be used to analyze water and water-suspended sediment containing from 1 to 50 μg/L of molybdenum. Samples containing more than 50 μg/L need to be diluted prior to chelation-extraction.

1.2 Suspended recoverable molybdenum is calculated by subtracting dissolved molybdenum from total recoverable molybdenum.

1.3 This method may be used to analyze bottom material containing at least 0.05 μg/g of molybdenum. Prepared sample solutions containing more than 50 μg/L of molybdenum need to be diluted.

1.4 Total recoverable molybdenum in water-suspended sediment needs to undergo preliminary digestion-solubilization by method I-3485, and recoverable molybdenum in bottom material needs to undergo preliminary digestion-solubilization by method I-5485 before being determined.

2. Summary of method

Molybdenum is determined by atomic absorption spectrometry following chelation with 8-hydroxyquinoline and extraction with methyl isobutyl ketone (MIBK). The extract is aspirated into a nitrous oxide-acetylene flame of the spectrometer (Chau and Lum-Shue- Chan, 1969).

3. Interferences

The method is free from interference from most elements commonly found in fresh water. Vanadium(V) and iron(III) enhance the absorption, and chromium(VI) and tungsten(VI) suppress it. With the addition of ascorbic acid, up to 50,000 μg/L of iron(III), 1,000 μg/L of vanadium(V), and 10,000 μg/L of chromium(VI) or tungsten(VI) can be tolerated (Chau and Lum-Shue-Chan, 1969).

4. Apparatus

4.1 *Atomic absorption spectrometer* equipped with electronic digital readout and automatic zero and concentration controls.

4.2 Refer to the manufacturer's manual to optimize instrument for the following:

Grating ------------ Ultraviolet
Wavelength -------- 313.3 nm
Source (hollow-cathode
lamp) ------------ Molybdenum
Oxidant ------------ Nitrous oxide
Fuel --------------- Acetylene
Type of flame ------- Fuel-rich

4.3 Different nitrous oxide burners may be used according to manufacturers' instructions.

5. Reagents

5.1 *Ascorbic acid solution*, 1 g/100 mL: Dissolve 1 g ascorbic acid in 100 mL demineralized water.

5.2 *Bromophenol blue indicator solution*, 0.1 g/100 mL: Dissolve 0.1 g bromophenol blue in 100 mL 50-percent ethanol.

5.3 *Hydrochloric acid*, 0.3M: Mix 25 mL concentrated HCl (sp gr 1.19) with demineralized water and dilute to 1 L.

5.4 *8-Hydroxyquinoline-methyl isobutyl ketone solution*, 1 g/100 mL: Dissolve 1 g 8-hydroxyquinoline in 100 mL MIBK. Prepare fresh daily.

5.5 *Methyl isobutyl ketone (MIBK)*.

5.6 *Molybdenum standard solution I,* 1.00 mL = 100 μg Mo: Dissolve 0.1500 g reagent-grade MoO₃ in 10 mL 0.1M NaOH (warm if necessary). Make just acidic with 0.1M HCl and dilute to 1,000 mL with demineralized water.

5.7 *Molybdenum standard solution II,* 1.00 mL = 1.0 μg Mo: Dilute 10.0 mL molybdenum standard solution I to 1,000 mL with demineralized water.

5.8 *Molybdenum standard solution III,* 1.0 mL = 0.10 μg Mo: Immediately before use, dilute 10.0 mL molybdenum standard solution II to 100 mL with demineralized water. This standard is used to prepare working standards at the time of analysis.

5.9 *Sodium hydroxide solution,* 2.5M: Dissolve 100 g NaOH in demineralized water and dilute to 1 L.

6. Procedure

6.1 Clean all glassware used in this determination with warm, dilute HNO₃ (1+9), and rinse with demineralized water immediately before use.

6.2 Pipet a volume of sample or prepared sample solution containing less than 5.0 μg Mo (100 mL max) into a 200-mL volumetric flask, and adjust the volume to approx 100 mL.

6.3 Prepare a demineralized-water blank with 1.5 mL concentrated. HNO₃ per liter of demineralized water and at least six standards, and adjust the volume of each to approx 100 mL with demineralized water.

6.4 Add 5 mL ascorbic acid solution and mix.

6.5 Add 2 drops bromophenol blue indicator solution and mix.

6.6 Adjust the pH by addition of 2.5M NaOH until a blue color persists. Add 0.3M HCl by drops until the blue color just disappears; then add 2.0 mL 0.3M HCl in excess. The pH at this point should be 2.3 (NOTE 1).

NOTE 1. The pH adjustment in paragraphs 6.5 and 6.6 may be made using a pH meter instead of using indicator. Add 2.5M NaOH to the solution until the pH is 2.3.

6.7 Add 5.0 mL 8-hydroxyquinoline-MIBK solution and shake vigorously for 15 min.

6.8 Allow the layers to separate; then add demineralized water until the ketone layer is completely in the neck of the flask.

6.9 While aspirating the ketone layer of the blank, use the automatic zero control to set the digital display to read zero concentration. While aspirating standards, use the automatic concentration control to set the digital display to read the concentration of the standards. Use at least six standards. Calibrate the instrument each time a set of samples is analyzed and check calibration at reasonable intervals.

7. Calculations

7.1 Determine the micrograms per liter of dissolved or total recoverable molybdenum in each sample from the digital display or printer. Dilute those samples containing molybdenum concentrations that exceed the working range of the method; repeat the chelation-extraction and multiply by the proper dilution factors.

7.2 To determine micrograms per liter of suspended recoverable molybdenum, subtract dissolved-molybdenum concentration from total-recoverable-molybdenum concentration.

7.3 To determine micrograms per gram of molybdenum in bottom-material samples, first determine the micrograms per liter of molybdenum as in paragraph 7.1; then

$$Mo\ (\mu g/g) = \frac{\mu g/L\ Mo \times \dfrac{mL\ of\ original\ digest}{1,000}}{wt\ of\ sample\ (g)}$$

8. Report

8.1 Report molybdenum, dissolved (01060), total recoverable (01062), and suspended recoverable (01061), concentrations as follows: less than 100 μg/L, nearest microgram per liter; 100 μg/L and above, two significant figures.

8.2 Report molybdenum, recoverable-from-bottom-material (01063), concentrations as follows: less than 10 μg/g, nearest 0.1 μg/g; 10 μg/g and above, two significant figures.

9. Precision

9.1 Precision for dissolved molybdenum for seven samples within the range of 1.3 to 56.7 μg/L may be expressed as follows:

$$S_T = 0.068X + 1.320$$

where

S_T = overall precision, micrograms per liter, and,

X = concentration of molybdenum, micrograms per liter.

The correlation coefficient is 0.8718.

9.2 Precision for dissolved molybdenum for four of the seven samples expressed in terms of the percent relative standard deviation is as follows:

Number of laboratories	Mean (μg/L)	Relative standard deviation (percent)
3	1.3	115
5	2.0	35
4	25.2	17
3	56.7	8

9.3 It is estimated that the percent relative standard deviation for total recoverable and suspended recoverable molybdenum and for recoverable molybdenum in bottom material will be greater than that reported for dissolved molybdenum.

Reference

Chau, Y. K., and Lum-Shue-Chan, K., 1969, Atomic absorption determination of microgram quantities of molybdenum in lake waters: Analytical Chimica Acta, v. 48, p. 205.

Molybdenum, atomic emission spectrometric, ICP

Parameter and Code:
Molybdenum, dissolved, I-1472-85 (μg/L as Mo): 01060

2. Summary of method

Molybdenum is determined simultaneously with several other constituents on a single sample by a direct-reading emission spectrometric method utilizing an induction-coupled argon plasma as an excitation source. See method I-1472, metals, atomic emission spectrometric, ICP.

297

Nickel, atomic absorption spectrometric, chelation-extraction

Parameters and Codes:
Nickel, dissolved, I-1500-85 (μg/L as Ni): 01065
Nickel, total recoverable, I-3500-85 (μg/L as Ni): 01067
Nickel, suspended recoverable, I-7500-85 (μg/L as Ni): 01066

1. Application

1.1 This method may be used to analyze water, brines, and water-suspended sediment containing from 1 to 100 μg/L of nickel. Sample solutions containing more than 100 μg/L need either to be diluted prior to chelation-extraction or to be analyzed by the atomic absorption direct method.

1.2 Suspended recoverable nickel is calculated by subtracting dissolved nickel from total recoverable nickel.

1.3 Total recoverable nickel in water-suspended sediment needs to undergo a preliminary digestion-solubilization by method I-3485 before being determined.

1.4 If the iron concentration of the sample solution exceeds 25,000 μg/L, determine nickel by the atomic absorption spectrometric direct method.

2. Summary of method

Nickel is determined by atomic absorption spectrometry following chelation with ammonium pyrrolidine dithiocarbamate (APDC) and extraction with methyl isobutyl ketone (MIBK). The extract is aspirated into the air-acetylene flame of the spectrometer (Fishman and Midgett, 1968).

3. Interferences

Concentrations of iron greater than 25,000 μg/L interfere by suppressing the nickel absorption.

4. Apparatus

4.1 *Atomic absorption spectrometer* equipped with electronic digital readout and automatic zero and concentration controls.

4.2 Refer to the manufacturer's manual to optimize instrument for the following:

Grating ------------ Ultraviolet
Wavelength -------- 232.0 nm
(NOTES 1 and 2)

Source (hollow-cathode lamp) ------------ Nickel
Oxidant ------------ Air
Fuel ---------------- Acetylene
Type of flame ------- Oxidizing

NOTE 1. Setting the proper wavelength for nickel is critical because of the presence of an ion line at 231.6 nm. By aspirating a standard that gives approx 50-percent absorption, the 232.0-nm absorption line can easily be set.

NOTE 2. The 352.4-nm wavelength, less susceptible to nonatomic absorbance, may be used. The resulting analytical curve is more nearly linear; however, the procedure reduces sensitivity.

4.3 Different burners may be used according to manufacturers' instructions.

5. Reagents

5.1 *Ammonium pyrrolidine dithiocarbamate solution*, 1 g/100 mL: Dissolve 1 g APDC in 100 mL demineralized water. Prepare fresh daily.

5.2 *Citric acid-sodium citrate buffer solution*: Dissolve 126 g citric acid monohydrate and 44 g sodium citrate dihydrate in demineralized water and dilute to 1 L with demineralized water. See NOTE 5 before preparation.

5.3 *Methyl isobutyl ketone* (MIBK).

5.4 *Nickel standard solution I*, 1.00 mL = 200 μg Ni: Dissolve 0.2000 g nickel powder in a minimum amount of dilute HNO_3. Heat to

increase rate of dissolution. Add 10.0 mL concentrated HNO_3 (sp gr 1.41) and dilute to 1,000 mL with demineralized water.

5.5 *Nickel standard solution II*, 1.00 mL = 2.00 μg Ni: Dilute 10.0 mL nickel standard solution I and 1 mL concentrated HNO_3 to 1,000 mL with demineralized water. This solution is used to prepare working standards at the time of analysis.

5.6 *Potassium hydroxide*, 10*M*: Dissolve 56 g KOH in demineralized water, cool, and dilute to 100 mL.

5.7 *Potassium hydroxide*, 2.5*M*: Dissolve 14 g KOH in demineralized water and dilute to 100 mL (NOTE 3).

NOTE 3. Alternatively, a 2.5*M* NH_4OH solution may be used. Add 167 mL concentrated NH_4OH (sp gr 0.90) to 600 mL demineralized water. Cool, and dilute to 1 L.

5.8 *Water, acidified*: Add 1.5 mL concentrated HNO_3 (sp gr 1.41) to 1 L of demineralized water.

6. Procedure

6.1 Clean all glassware used in this determination with warm, dilute HNO_3 (1+9) and rinse with demineralized water immediately before use.

6.2 Pipet a volume of sample solution containing less than 10 μg Ni (100 mL max) into a 200-mL volumetric flask, and adjust the volume to approx 100 mL.

6.3 Prepare a blank of acidified water and sufficient standards, and adjust the volume of each to approx 100 mL with acidified water.

6.4 With a pH meter, adjust the pH of each solution to 2.4 with 2.5*M* KOH (NOTES 4 and 5). Shake for 3 min.

NOTE 4. For water-suspended sediment samples that have been digested, add 1 to 2 mL 10*M* KOH or concentrated NH_4OH (sp gr 0.90) before pH adjustment.

NOTE 5. If an automated titration system is used to adjust the pH, add 2.5 mL citric acid-sodium citrate buffer solution prior to pH adjustment. This will prevent over-shooting the end point in poorly buffered samples.

6.5 Add 2.5 mL APDC solution and mix.

6.6 Add 10.0 mL MIBK and shake vigorously for 3 min.

6.7 Allow the layers to separate and add demineralized water until the ketone layer is completely in the neck of the flask.

6.8 Aspirate the ketone layer within 1 h. Aspirate the ketone layer of the blank to set the automatic zero control. Use the automatic concentration control to set the concentrations of standards. Use at least six standards. Calibrate the instrument each time a set of samples is analyzed and check calibration at reasonable intervals.

7. Calculations

7.1 Determine the micrograms per liter of dissolved or total recoverable nickel in each sample from the digital display or printer while aspirating each sample. Dilute those samples containing concentrations of nickel that exceed the working range of the method; repeat the chelation-extraction and multiply by the proper dilution factors.

7.2 To determine the micrograms per liter of suspended recoverable nickel, subtract dissolved-nickel concentration from total-recoverable-nickel concentration.

8. Report

Report nickel, dissolved (01065), total-recoverable (01067), and suspended-recoverable (01066), concentrations as follows: less than 10 μg/L, nearest microgram per liter; 10 μg/L and above, two significant figures.

9. Precision

9.1 The standard deviation for dissolved nickel within the range of 3.8 to 23.2 μg/L for 20 samples was found to be independent of concentration. The 95-percent confidence interval for the average standard deviation of 4.3 μg/L ranged from 3.8 to 5.0 μg/L.

9.2 Precision for dissolved nickel for five of the 20 samples expressed in terms of the percent relative standard deviation is as follows:

Number of laboratories	Mean (μg/L)	Relative standard deviation (percent)
5	3.8	11
5	4.4	61
4	10.2	38
8	10.5	16
13	23.2	23

9.3 It is estimated that the percent relative standard deviation for total-recoverable and suspended recoverable nickel will be greater than that reported for dissolved nickel.

Reference

Fishman, M. J., and Midgett, M. R., 1968, Extraction techniques for the determination of cobalt, nickel, and lead in fresh water by atomic absorption, *in* Trace inorganics in water: American Chemical Society Advances in Chemistry Series, no. 73, p. 230–5.

Nickel, atomic absorption spectrometric, direct

Parameters and Codes:
Nickel, dissolved, I-1499-85 (μg/L as Ni): 01065
Nickel, total recoverable, I-3499-85 (μg/L as Ni): 01067
Nickel, suspended recoverable, I-7499-85 (μg/L as Ni): 01066
Nickel, recoverable-from-bottom-material, dry wt, I-5499-85 (μg/g as Ni): 01068

1. Application

1.1 This method may be used to analyze water and water-suspended sediment containing at least 100 μg/L of nickel. Sample solutions containing more than 1,000 μg/L need either to be diluted or to be read on a less expanded scale. Sample solutions containing less than 100 μg/L and brines need to be analyzed by the atomic absorption spectrometric chelation-extraction method, providing that the interference limits discussed in that method are not exceeded.

1.2 Suspended recoverable nickel is calculated by subtracting dissolved nickel from total recoverable nickel.

1.3 This method may be used to analyze bottom material containing at least 5.0 μg/g of nickel. Sample solutions containing more than 1,000 μg/L need either to be diluted or to be read on a less expanded scale.

1.4 Total recoverable nickel in water-suspended sediment needs to undergo preliminary digestion-solubilization by method I-3485, and recoverable nickel in bottom material needs to undergo preliminary digestion-solubilization by method I-5485 before being determined.

2. Summary of method

Nickel is determined by atomic absorption spectrometry by direct aspiration of the sample into an air-acetylene flame without preconcentration or pretreatment.

3. Interferences

Individual concentrations of sodium (9,000 mg/L), potassium (9,000 mg/L), calcium (4,000 mg/L), magnesium (4,000 mg/L), sulfate (9,000 mg/L), and cadmium, lead, copper, zinc, cobalt, and chromium (10,000 μg/L each) do not interfere. Higher concentrations of each constituent were not investigated.

4. Apparatus

4.1 *Atomic absorption spectrometer* equipped with electronic digital readout and automatic zero and concentration controls.

4.2 Refer to the manufacturer's manual to optimize instrument for the following:

Grating ------------ Ultraviolet
Wavelength -------- 232.0 nm
(NOTES 1 and 2)
Source (hollow-cathode lamp) ------------ Nickel
Oxidant ------------ Air
Fuel ---------------- Acetylene
Type of flame ------- Oxidizing

NOTE 1. Setting the proper wavelength for nickel is critical because of the presence of an ion line at 231.6 nm. By aspirating a standard that gives approx 50-percent absorption, the 232.0 nm-absorption line can easily be set.

NOTE 2. The 352.4-nm wavelength, less susceptible to nonatomic absorbance, may be used. The resulting analytical curve is more nearly linear; however, the procedure reduces sensitivity.

5. Reagents

5.1 *Nickel standard solution I*, 1.00 mL = 200 μg Ni: Dissolve 0.2000 g Ni powder in a minimum amount of dilute HNO_3. Heat to increase rate of dissolution. Add 10.0 mL

concentrated HNO_3 (sp gr 1.41) and dilute to 1,000 mL with demineralized water.

5.2 *Nickel standard solution II*, 1.00 mL = 20 μg Ni: Dilute 100 mL nickel standard solution I and 1 mL concentrated HNO_3 (sp gr 1.41) to 1,000 mL with demineralized water.

5.3 *Nickel standard working solutions*: Prepare a series of at least six standard working solutions containing from 100 to 1,000 μg/L of nickel by appropriate dilution of nickel standard solution II with acidified water. Prepare fresh daily.

5.4 *Water, acidified*: Add 1.5 mL concentrated HNO_3 (sp gr 1.41) to 1 L of demineralized water.

6. Procedure

Aspirate the blank (acidified water) to set the automatic zero control. Use the automatic concentration control to set the concentrations of standards. Use at least six standards. Calibrate the instrument each time a set of samples is analyzed and check calibration at reasonable intervals.

7. Calculations

7.1 Determine the micrograms per liter of dissolved or total recoverable nickel in each sample from the digital display or printer while aspirating each sample. Dilute those samples containing concentrations of nickel that exceed the working range of the method and multiply by the proper dilution factors.

7.2 To determine micrograms per liter of suspended recoverable nickel, subtract dissolved-nickel concentration from total-recoverable-nickel concentration.

7.3 To determine micrograms per gram of nickel in bottom-material samples, first determine the micrograms per liter of nickel as in paragraph 7.1; then

$$Ni\ (\mu g/g) = \frac{\mu g/L\ Ni \times \dfrac{mL\ of\ original\ digest}{1,000}}{wt\ of\ sample\ (g)}$$

8. Report

8.1 Report nickel, dissolved (01065), total-recoverable (01067), and suspended- recoverable

(01066), concentrations to the nearest 100 μg/L.

8.2 Report nickel, recoverable-from-bottom-material (01068), concentrations as follows: less than 100 μg/g, nearest 10 μg/g; 100 μg/g and above, two significant figures.

9. Precision

9.1 Precision for dissolved nickel for 16 samples within the range of 6.2 to 36.2 μg/L (NOTE 3) may be expressed as follows:

$$S_T = 0.431X + 2.88$$

where

S_T = overall precision, micrograms per liter, and

X = concentration of nickel, micrograms per liter.

The correlation coefficient is 0.7593.

NOTE 3. Precision data for nickel are below the reporting level of 100 μg/L. Samples were not available that contained greater nickel concentrations; however, precision should improve at greater concentrations.

9.2 Precision for dissolved nickel for five of the 16 samples expressed in terms of the percent relative standard deviation is as follows:

Number of laboratories	Mean (μg/L)	Relative standard deviation (percent)
5	6.2	18
11	10.5	80
6	10.8	15
6	20.0	46
19	36.2	47

9.3 It is estimated that the percent relative standard deviation for total recoverable and suspended recoverable nickel and for nickel in bottom material will be greater than that reported for dissolved nickel.

9.4 Precision for total recoverable nickel expressed in terms of the percent relative standard for two water-suspended sediments is as follows:

Number of laboratories	Mean (μg/L)	Relative standard deviation (percent)
10	26.2	58
10	35.9	53

Nickel, atomic absorption spectrometric, graphite furnace

Parameter and Code:
Nickel, dissolved, I-1501-85 (µg/L as Ni): 01065

1. Application

1.1 This method may be used to determine nickel in low ionic-strength water and precipitation. With deuterium background correction and a 20-µL sample, the method is applicable in the range from 1 to 100 µg/L. With Zeeman background correction and a 20-µL sample, the method is applicable in the range from 1 to 80 µg/L. Sample solutions that contain nickel concentrations exceeding the upper limits must be diluted or preferably be analyzed by the atomic absorption spectrometric direct or chelation-extraction method.

1.2 The analytical range and detection limits can be increased or possibly decreased by varying the volume of sample injected or the instrumental settings. Purification of reagents and use of ASTM Type 1 water (Method D-1193, American Society for Testing and Materials, 1984) may result in lower detection limits.

2. Summary of method

Nickel is determined by atomic absorption spectrometry in conjunction with a graphite furnace containing a graphite platform (Hinderberger and others, 1981). A sample is placed on the graphite platform, and the sample is then evaporated to dryness, charred, and atomized using high-temperature ramping. The absorption signal generated during atomization is recorded and compared with standards.

3. Interferences

3.1 Interferences in low ionic-strength samples, such as precipitation, normally are quite low. In addition, the use of the graphite platform reduces the effects of many interferences. Calcium (25 mg/L), magnesium (8 mg/L), sodium (20 mg/L), sulfate (34 mg/L), and chloride (25 mg/L) do not interfere. Higher concentrations of these constituents were not investigated.

3.2 Precipitation samples usually contain very low concentrations of nickel. Special precautionary measures must be employed during both sample collection and laboratory determination to prevent contamination.

4. Apparatus

4.1 *Atomic absorption spectrometer*, for use at 232.0 nm and equipped with background correction, digital integrator to quantitate peak areas, graphite furnace with temperature programmer, and automatic sample injector. The programmer must have high-temperature ramping and stopped-flow capabilities.

4.1.1 Refer to the manufacturer's manual to optimize instrumental performance. The analytical ranges reported in paragraph 1.1 are for a 20-µL sample (NOTE 1).

NOTE 1. A 20-µL sample generally requires 30 s to dry. Samples that have a complex matrix may require a longer drying and charring time.

4.1.2 *Graphite furnace*, capable of reaching temperatures sufficient to atomize the element of interest. Warning: dial settings frequently are inaccurate and newly conditioned furnaces require temperature calibration.

4.1.3 *Graphite tubes and platforms.* Pyrolytically coated graphite tubes and solid pyrolytic graphite platforms are recommended.

4.2 *Labware.* Many trace metals at very low concentrations have been found to sorb very rapidly to glassware. To preclude this, fluorinated ethylene propylene (FEP) or Teflon labware may be used. Alternately, glassware, particularly flasks and pipets, may be treated with silicone anti-wetting agent such as Surfacil

(Pierce Chemical Co., Rockford, IL, 61105) according to the manufacturer's instructions. Autosampler cups must be checked for contamination. Lancer (1831 Olive St., St. Louis, MO, 63103) polystyrene disposable cups have been found to be satisfactory after acid rinsing. Alternately, reuseable Teflon or FEP cups may be used.

4.3 *Argon*, standard, welder's grade, commercially available. Nitrogen may also be used if recommended by the instrument manufacturer.

5. Reagents

5.1 *Nickel standard solution I*, 1.00 mL = 1,000 μg Ni: Dissolve 1.0000 g Ni powder in a minimum of dilute HNO_3. Heat to increase rate of dissolution. Add 10 mL high-purity, concentrated HNO_3 (sp gr 1.41), Ultrex or equivalent, and dilute to 1,000 mL with Type 1 water.

5.2 *Nickel standard solution II*, 1.00 mL = 10.0 μg Ni: Dilute 10.0 mL nickel standard solution I to 1,000 mL (NOTE 2).

NOTE 2. Use acidified Type 1 water (paragraph 5.6) to make dilutions. All standards must be stored in sealed Teflon or FEP containers. Each container must be rinsed twice with a small volume of standard before being filled. Standards stored for 6 months in FEP containers yielded values equal to those of freshly prepared standards.

5.3 *Nickel standard solution III*, 1.00 mL = 1.00 μg Ni: Dilute 100.0 mL nickel standard solution II to 1,000 mL. This standard is used to prepare working standards serially at time of analysis.

5.4 *Nickel standard solution IV*, 1.00 mL = 0.010 μg Ni: Dilute 10.0 mL nickel standard solution III to 1,000 mL. This standard also is used to prepare working standards serially at time of analysis.

5.5 *Nitric acid, concentrated*, high-purity, (sp gr 1.41): J. T. Baker "Ultrex" brand HNO_3 has been found to be adequately pure; however, each lot must be checked for contamination. Analyze acidified Type 1 water for nickel. Add an additional 1.5 mL of concentrated HNO_3/liter of water, and repeat analysis. The integrated signal should not increase by more than 0.001 absorbance-seconds.

5.6 *Water, acidified*, Type 1: Add 1.5 mL high purity concentrated HNO_3 (sp gr 1.41) to each liter of water.

5.7 *Water*, Type 1.

6. Procedure

6.1 Systematically clean and rinse work areas with deionized water on a regular schedule. Use a laminar flow hood or a "clean room" environment during sample transfers. Ideally, the autosampler and the graphite furnace should be in a clean environment.

6.2 Soak autosampler cups at least overnight in a 1+1 solution of Type 1 water and high-purity nitric acid.

6.3 Rinse the sample cups twice with sample before filling. Place cups in sample tray and cover. Adjust sampler so that only the injection tip contacts the sample.

6.4 In sequence, inject 20-μL aliquots of blank and working standards, and analyze. Analyze the blank and working standards twice. Construct the analytical curve from the integrated peak areas (absorbance-seconds). Generally, the curve should be linear to a peak-absorbance (peak-height) value of 0.40 absorbance units.

6.5 Similarly, inject and analyze the samples twice. Every tenth sample cup should contain either a standard or a reference material.

6.6 Restandardize as required. Minor changes of values for known samples usually indicate deterioration of the furnace tube, contact rings, and (or) platform. A major variation usually indicates either autosampler malfunction or residue buildup from a complex matrix in a previous sample.

7. Calculations

Determine the micrograms per liter of nickel in each sample from the digital display or printer output. Dilute those samples containing concentrations of nickel that exceed the working range of the method; repeat the analysis, and multiply by the proper dilution factors.

8. Report

Report nickel, dissolved (01065), concentrations as follows: less than 10 μg/L, nearest 1 μg/L; 10 μg/L and above, two significant figures for both deuterium background correction and Zeeman background correction.

9. Precision

9.1 Analysis of two samples by a single operator using deuterium background correction is as follows:

Number of replicates	Mean (μg/L)	Standard deviation (μg/L)	Relative standard deviation (percent)
10	5.8	0.6	10.3
5	9.8	.8	8.2

9.2 Analysis of three samples six times each by a single operator using Zeeman background correction is as follows:

Mean (μg/L)	Standard deviation (μg/L)	Relative standard deviation (percent)
14.5	0.6	4.1
48.3	.5	1.0
79.7	.5	.6

9.3 The precision and bias for the Zeeman background correction were tested on deionized water and tap water (specific conductance 280 μS/cm). A known amount of nickel was added to each sample, and single-operator precision and bias for six replicates are as follows:

Amount added (μg/L)	Amount found (μg/L)	Standard deviation (μg/L)	Relative standard deviation (percent)	Recovery (percent)
Deionized water				
4.35	4.0	0.6	16	92
8.7	9.3	.5	5	107
13	12.3	.8	7	95
26	24.5	1.6	6	94
27.5	22.8	1.5	7	83
Tap water (NOTE 3)				
4.35	4.2	.8	15	97
8.7	9.6	.8	7	110
13	12.0	1.0	7	92
26	21.5	1.5	7	83
27.5	24.1	1.9	8	88

NOTE 3. The tap water contained 1.2 μg/L of nickel, and the standard deviation and percent relative standard deviation were calculated prior to subtraction of nickel originally present.

9.4 The precision and bias for the deuterium background method were tested on deionized water and tap water (specific conductance 280 μS/cm). A known amount of nickel was added to each sample, and single-operator precision and bias for six replicates are as follows:

Amount added (μg/L)	Amount found (μg/L)	Standard deviation (μg/L)	Relative standard deviation (percent)	Recovery (percent)
Deionized water				
4.35	3.8	1.0	26.3	87
8.7	9.0	.6	6.7	103
13	13.2	1.2	9.1	102
26	25.5	3.1	12.2	98
27.5	23.2	1.7	7.3	84
Tap water (NOTE 3)				
4.35	5.7	.8	11.6	131
8.7	9.8	.6	5.5	113
13	12.8	1.3	9.3	98
26	22.5	3.1	13.0	87
27.5	24.1	2.7	10.9	88

9.5 The standard deviation from interlaboratory data, without regard to type of background correction and use of matrix modifiers, if any, for dissolved nickel within the range of 3.8 to 22.5 μg/L for 16 samples was found to be independent of concentration. The 95-percent confidence interval for the average standard deviation of 5.2 μg/L ranged from 4.5 to 6.1 μg/L.

References

American Society for Testing and Materials, 1984, Annual book of ASTM standards, section 11, water: Philadelphia, American Society for Testing and Materials, v. 11.01, p. 39–41.

Cooksey, M., and Barnett, W. B., 1979, Matrix modification and the method of additions in flameless atomic absorption: Atomic Absorption Newsletter, v. 18, p. 101–5.

Fernandez, F. J., Beatty, M. M., and Barnett, W. B., 1981, Use of the L'vov platform for furnace atomic absorption applications: Atomic Spectroscopy, v. 2, p. 16–21.

Hinderberger, E. J., Kaiser, M. L., and Koirtyohann, S. R., 1981, Furnace atomic absorption analysis of biological samples using the L'vov platform and matrix modification: Atomic Spectroscopy, v. 2, p. 1–11.

Manning, D. C., and Slavin, W., 1983, The determination of trace elements in natural waters using the stabilized temperature platform furnace: Applied Spectroscopy, v. 37, p. 1–11.

Ottaway, J. M., 1982, A revolutionary development in graphite furnace atomic absorption: Atomic Spectroscopy, v. 3, p. 89–92.

Slavin, W., Carnrick, G. R., and Manning, D. C., 1982, Magnesium nitrate as a matrix modifier in the stabilized temperature platform furnace: Analytical Chemistry, v. 54, p. 621–4.

Nickel, total-in-sediment, atomic absorption spectrometric, direct

Parameter and Code:
Nickel, total, I-5474-85 (mg/kg as Ni): none assigned

2. Summary of method

A sediment sample is dried, ground, and homogenized. The sample is digested with a combination of nitric, hydrofluoric, and perchloric acids in a Teflon beaker heated on a hotplate at 200 °C. Nickel is determined on the resulting solution by atomic absorption spectrometry. See method I-5474, metals, major and minor, total-in-sediment, atomic absorption spectrometric, direct.